



# Ceramic Matrix Composites (CMC) Life Prediction Method Development

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## **SUMMARY**

Advanced launch systems (e.g., Reusable Launch Vehicle and other Shuttle Class concepts, Rocket-Based Combine Cycle, etc.), and interplanetary vehicles will very likely incorporate fiber reinforced ceramic matrix composites (CMC) in critical propulsion components. The use of CMC is highly desirable to save weight, to improve reuse capability, and to increase performance. CMC candidate applications are mission and cycle dependent and may include turbopump rotors, housings, combustors, nozzle injectors, exit cones or ramps, and throats. For reusable and single mission uses, accurate prediction of life is critical to mission success. The tools to accomplish life prediction are very immature and not oriented toward the behavior of carbon fiber reinforced silicon carbide (C/SiC), the primary system of interest for a variety of space propulsion applications. This paper describes an approach to satisfy the need to develop an integrated life prediction system for CMC that addresses mechanical durability due to cyclic and steady thermomechanical loads, and takes into account the impact of environmental degradation.

## INTRODUCTION

Current state-of-the art CMC life prediction methodologies embodied in NASALife (ref. 1) and similar codes are based on empirical formulations. In general, these have to be calibrated using experimental data. A shortcoming of these approaches is that any kind of changes in fiber architecture, constituent volume ratios, or other variables make the material system completely “new.” This requires that the empirical relations be recalibrated by extensive additional experimental testing. Much of this additional cost and time can be reduced if the analytical models are based on micromechanics. Once calibrated for a specific CMC system, the predictive capability of the model can then be utilized without additional calibration. NASALife was developed under the Enabling Propulsion Materials Project of the High Speed Research Program. Development of these codes has focused on material systems that are markedly different from carbon fiber reinforced silicon carbide. These approaches are lacking because they are not physics-based for accurate prediction of damage due to fatigue and fracture loading conditions. They also do not account for environmental effects due to water vapor attack of silica oxide scales and carbon oxidation, which are expected to be major factors in the application of C/SiC to space propulsion systems. Thus, current methods, and the underlying empirical equations upon which they are based, are inadequate for predicting the reusable life of C/SiC space propulsion hardware. The approach outlined in this paper is designed to resolve these shortcomings.

## APPROACH AND STATUS

The overall effort focuses on providing a robust life prediction methodology that will allow confident determination of the reusable life capability of C/SiC space propulsion hardware (fig. 1). For the reasons outlined in figure 2, standard C/SiC (T-300 fibers, SiC seal coat) from Honeywell Advanced Composites, Inc. was chosen as the baseline material for this study. This will be accomplished by enhancing NASALife to capture the damage and degradation mechanisms associated with static and cyclic thermal and mechanical loading of C/SiC components in a high temperature, high pressure, steam containing environment (figs. 3 and 4). Also, approaches for life extension will be sought.

The reaction of silica scales with water vapor is the most straightforward aspect of the environmental attack problem to characterize and model because stress state interactions are insignificant. Current state of the art consists of both experimental data and a model for SiC and  $\text{Si}_3\text{N}_4$  recession due to formation of volatile silicon hydroxides in combustion conditions typical of aircraft engines (figs. 5 and 6) (ref. 2). The model predicts material recession rates as a function of water vapor partial pressure, total pressure, gas velocity, and material temperature. In this task the model is being extended to pressures, gas chemistries, gas velocities, and material temperatures typical of the rocket engine environment (figs. 7 and 8). High pressure, low velocity tests will be run upstream of the nozzle throat at various  $\text{H}_2/\text{O}_2$  mixture ratios. Atmospheric pressure, high velocity tests will be run at various mixture ratios downstream of the throat.

The second aspect of the environmental attack problem arises because C/SiC composites have a microcracked SiC matrix in the as-produced condition (fig. 9). As a result, the carbon coating on the fibers and the carbon fibers themselves are subject to oxidation attack when the cracks are opened (refs. 3 and 4). This degradation mechanism occurs at temperatures below the composite fabrication temperature under zero stress conditions (fig. 10), and at all elevated temperatures sufficient for oxidation of the fibers when stress is applied (fig. 11). Since oxidizing conditions are expected to be present in the service environment of most C/SiC components, prediction of oxidation attack is a key ingredient of the life prediction model. A more thorough understanding of the effects of environment, temperature, and stress on the degradation of carbon fibers is being developed so that material limitations can be better identified and methods of improving oxidation resistance can be addressed. The development of a fiber oxidation model is being pursued (figs. 12 and 13). The model is physics and experimentally based. It incorporates such variables as reaction rate, diffusion coefficient, temperature, partial pressure, and environment. It tracks the recession of an array of fibers in a cracked matrix so that the oxidation kinetics involved in carbon fiber degradation can be studied. Stress rupture tests conducted will aid in the development of the model.

Physics based, probabilistic lifting models are being pursued. The models will address issues inherently related to composite materials—stochastic characterization of strength, life, and orthotropic material response. Experimental stress rupture and fatigue testing will be carried out in appropriate environments in support of model calibration and validation. Additional testing will be done for the characterization of the mechanical behavior of advanced ceramic composites proposed for use in space propulsion engine components, such as nozzle structures and turbo-

machinery. The lifting models developed will be implemented in NASALife. A parallel effort for a micro-mechanics (fiber/coating/matrix) based approach to predict stiffness, strength, and life at the coupon level is also being pursued (refs. 5 to 7). Current on-going research tasks have led to a library of computer codes (CEMCAN/WCMC, PCGINA) developed specifically for the design of CMC. These computer codes will be adapted to C/SiC to provide state of the art design tools.

Lifting schemes, such as those contained within NASALife and currently employed for CMCs, are adapted from models originally developed for design with metals. These traditional models are comprised of modified Miner's rules, rain-flow calculations, empirical knockdown factors, safety factors, etc. Under the current research program, a probabilistic residual strength model is being pursued. Residual strength is taken as the damage metric for stress rupture and mechanical fatigue life models. Initial static strength, intermediate residual strength, and time or cycles to failure are all treated as random variables (see fig. 14). In addition, efforts are underway to develop physics based models at the fiber/matrix level for life determination, and environmental effects. In the mean time, the residual strength model utilizes empirical relationships where needed, but is open to modification and incorporation of new models, such as micromechanical models and models for environmental degradation, as they become available.

The test matrix for tensile, creep-rupture, and fatigue testing was formulated to satisfy several requirements: (1) Calibration and verification of the probabilistic residual strength model. (2) Assessment of usable service conditions (i.e., temperature, stress, and environment) for C/SiC. (3) To determine the effect of alternative fiber architecture on material behavior and model capability (fig. 15). The initial stress-rupture data generated are shown in figure 16. Tests were conducted in six different environments, using a temperature of 1200 °C and stress of 83 MPa (10 ksi) for all tests. Similar lives were obtained for specimens tested in air and environments containing steam, while specimens tested in vacuum did not fail. These data are consistent with the SiC recession (fig. 5), TGA (fig. 10), and stressed oxidation data (fig. 11), indicating that the environment plays a key role in the high temperature performance of C/SiC.

Methods to limit environmental attack by oxidation and reaction with water vapor are being developed. The proposed work will explore three ways to protect the integrity of the C/SiC composite: (1) external barrier coatings, (2) additives and pretreatment to promote oxide sealing of preexisting cracks and those that form in service, and (3) interphase coatings to protect the carbon fiber from oxidation.

## CONCLUDING REMARKS

Life prediction for C/SiC is a complex problem involving many interactive mechanisms. The plan outlined here will analyze mechanisms in isolation as well as the interactions, develop mechanistic lifting models, understand the importance of statistics in C/SiC behavior, and develop methods to extend C/SiC life.

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- Primary goal:
  - Develop and verify a robust methodology for confident determination of the reusable life capability of C/SiC space propulsion hardware.

- Secondary goals:
  - To ground the methodology with mechanism-based descriptions of mechanically and environmentally induced damage.
  - To expand the database for C/SiC.
  - To identify methods for life enhancement.
  - To directly support flight experiments which use CMC propulsion components.

- Positives
  - Reproducible
  - Readily available
  - Realistic set of life limiting mechanisms
  - Controllable life
  - Of real interest

- Negative
  - Costly

Figure 1.—Program objectives.

Figure 2.—Standard ACI C/SiC chosen as model material.

- Environmental
  - Surface recession due to moisture
  - Interface and fiber oxidation
- Mechanical
  - Strains due to thermal and mechanical loads
  - Cycling of loads (LCF, HCF)
  - Creep

Figure 3.—C/SiC life controlled by complex, interactive mechanisms.

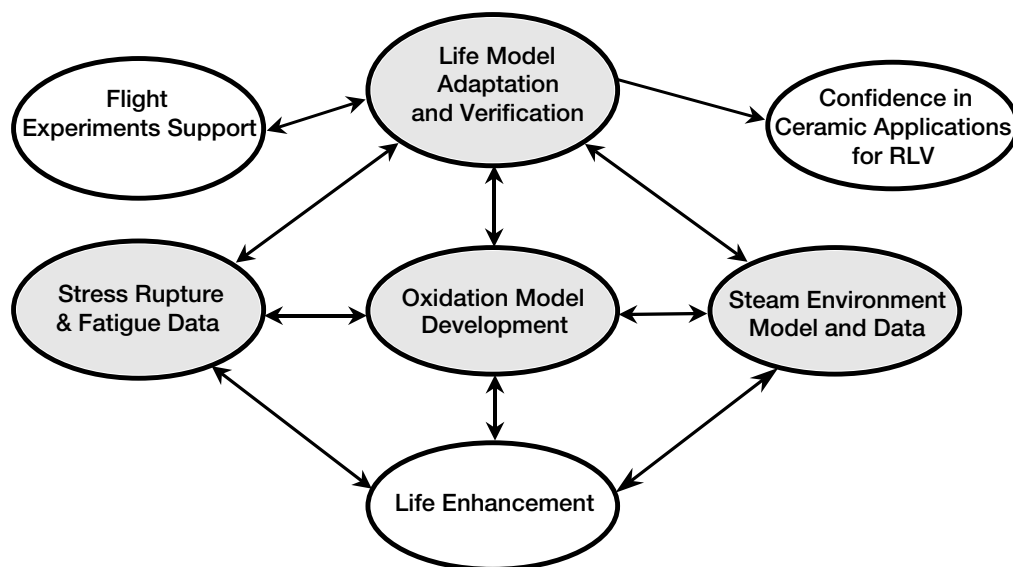


Figure 4.—C/SiC life prediction task organization.



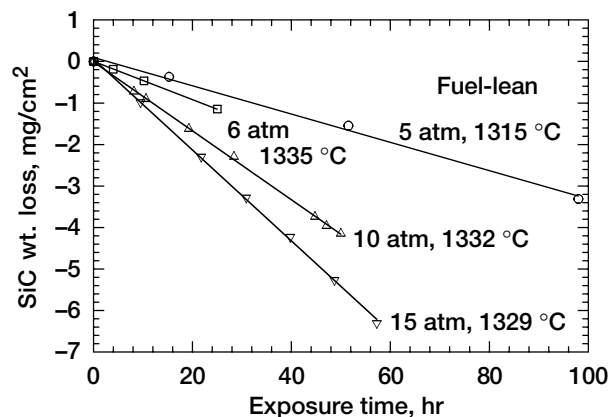
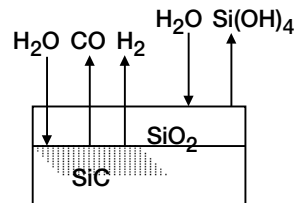
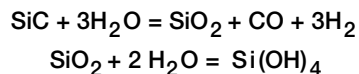


Figure 5.—Pressure dependence of SiC recession in combustion environments.



• Chemical model for  $\text{Si(OH)}_4$ :

$$k_1 \sim \exp(-57 \text{ kJ/mol/RT}) P^{1.50} v^{0.50}$$

Figure 6.—SiC volatilization mechanism in fuel-lean combustion environments.

- Extend model for silica volatilization (SiC recession) to pressures, gas chemistries, gas velocities, and material temperatures typical of rocket engine environments.
- Verify with materials tests in a rocket engine environment.
  - O/F (oxygen to fuel) ratio
  - gas pressures
  - gas velocities
  - material temperatures

Figure 7.—Steam environment model and data.

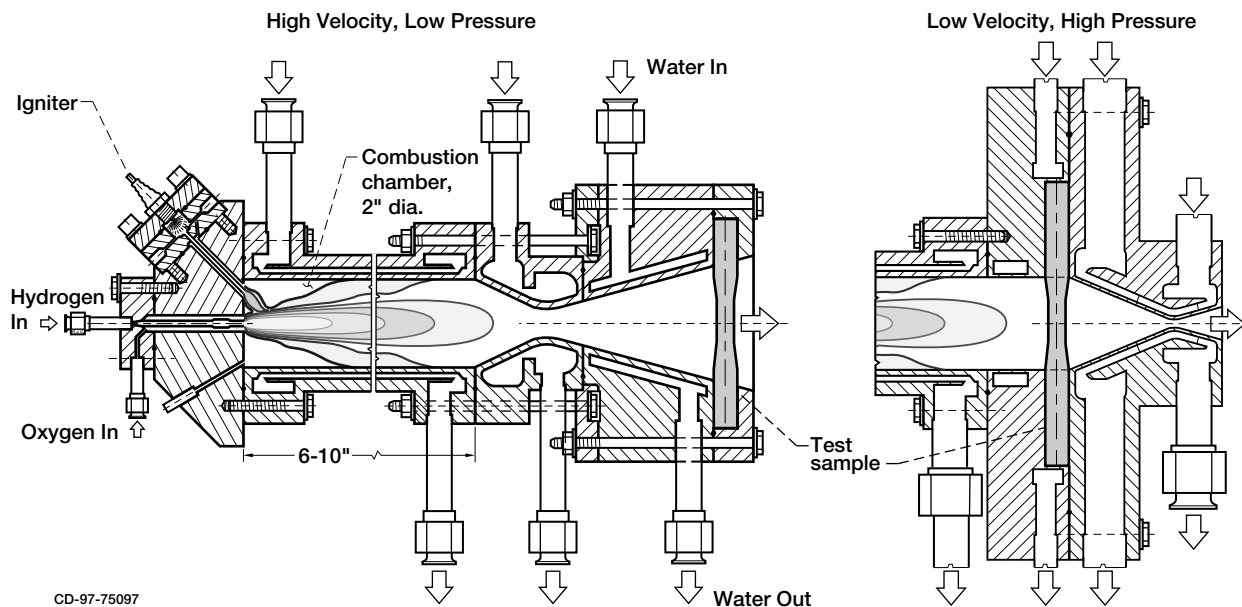
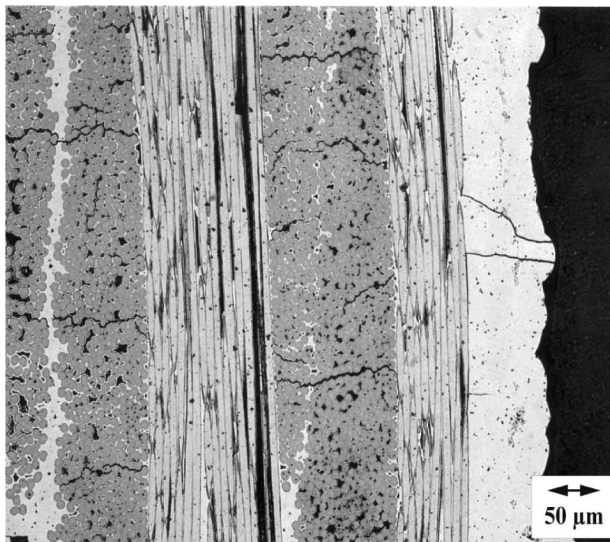
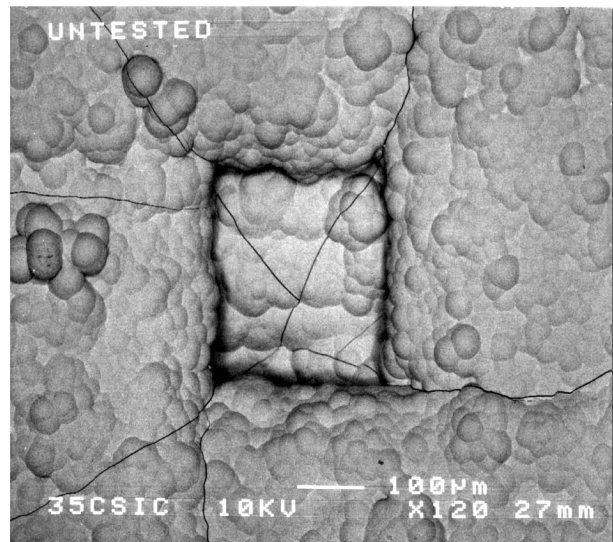


Figure 8.—Hydrogen/oxygen combustion test stand configurations.

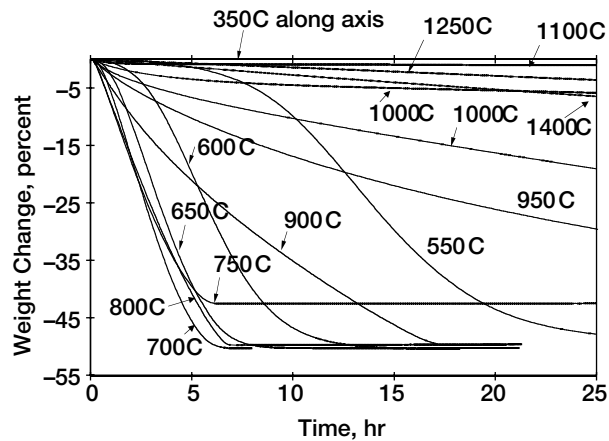


- Internal cracking contained with [90] plies and in seal coating



- Surface cracking of CVI seal coating

Figure 9.—As-manufactured microstructure of ACI C/SiC (C/SiC has internal cracking due to fabrication).



Samples: 1" x 0.5" TGA C/SiC coupon bars (~2.4 g)  
 Fibers only exposed to oxygen at cracked regions  
 Complete fiber burnout at 650 to 800 °C after 5 hr  
 Crack closure at > 800 °C  
 Minimal consumption at temperatures around  
 1100 °C after 25 hr

Figure 10.—Oxidation of C/SiC coupons in a TGA.

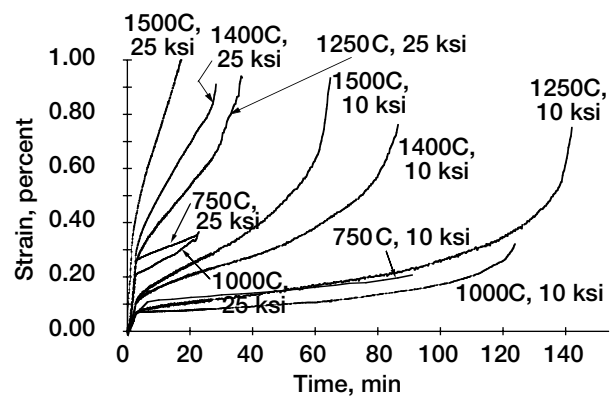


Figure 11.—Stressed oxidation of C/SiC.

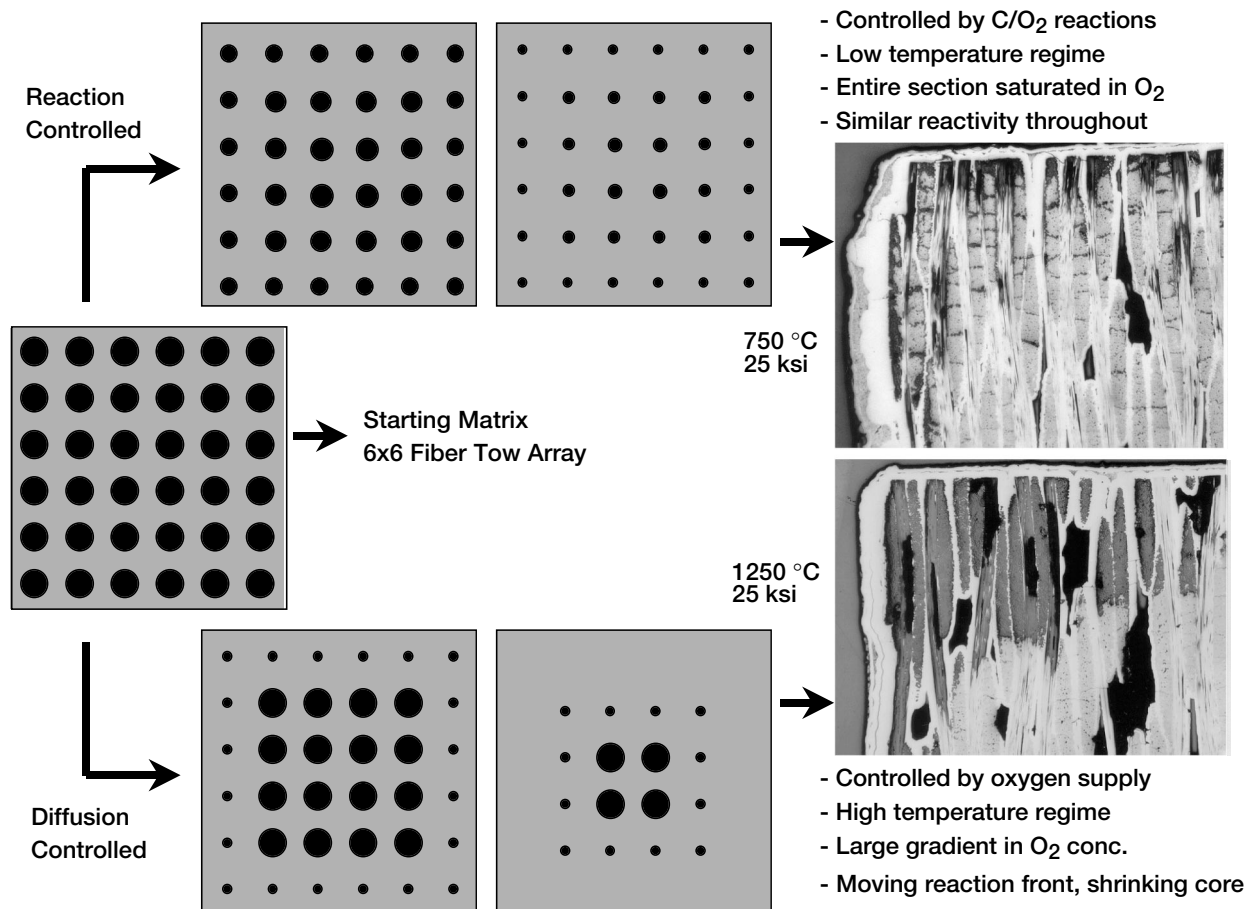


Figure 12.—Model development for prediction of C/SiC strength loss due to oxidation of carbon fiber.

- Advance the development of a model that predicts degradation of an array of carbon fibers in a cracked ceramic matrix.
- Determine the role that temperature, stress, and environment play in the oxidation rate of carbon fibers through experimentation (analysis of stress rupture tests) and analysis in the model.
- Develop a correlation between composite strength/failure and carbon consumption so the model can be used to predict the life of the CMC material under application conditions.

Figure 13.—Oxidation model development.

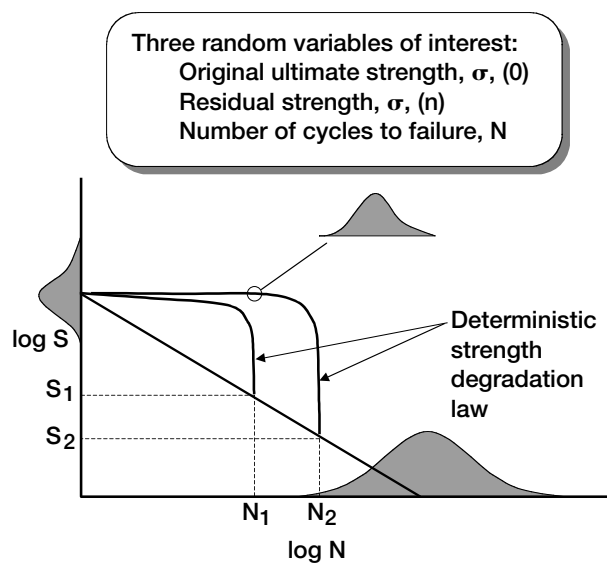


Figure 14.—Probabilistic model development for C/SiC.

### 1. Material Design Limitation

- Creep-rupture in air, PO<sub>2</sub> and vacuum (determine PO<sub>2</sub> for model testing).
- Creep-rupture in humidity (determine material behavior in steam).
- Creep-rupture in PO<sub>2</sub> on narrow & wide specimens (assess life dependency on specimen area)

### 2. Durability Model Calibration & Verification

(in partial pressure of O<sub>2</sub>).

- Tensile tests - 24 per temperature.
- Creep-rupture tests - 60 per temperature.
  - 20 tested to failure (calibration).
  - 20 stopped prior to failure and residual strength (verification).
  - 20 to failure (verification).

### 3. Fatigue Model Calibration, & Verification

(in partial pressure of O<sub>2</sub>).

- Fatigue tests - 60 per temperature.
  - 20 tested to failure (calibration).
  - 20 stopped prior to failure and residual strength (verification).
  - 20 to failure (verification).
- Feature tests - specimens with holes, notches, etc. (benchmark model predictive capability).
- Fatigue or creep rupture of alternate fiber architecture (calibration).

Figure 15.—Test matrix.

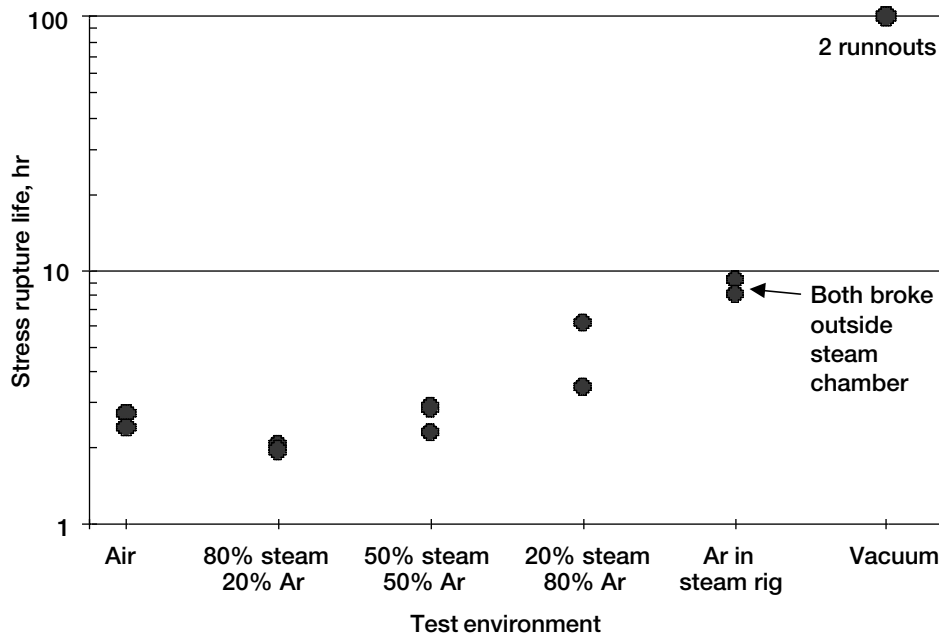


Figure 16.—Stress-rupture lives for [0/90] C/SiC (tests conducted at 1200 °C, 10 ksi).

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